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IS 5449 (1969): Methods for determination of water soluble chromates in textile materials [TXD 5: Chemical Methods of Test]



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METHODS FOR DETERMINATION OF WATER SOLUBLE CHROMATE IN TEXTILE MATERIALS

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG

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Indian Standard

METHODS FOR DETERMINATION OF WATER SOLUBLE CHROMATE IN TEXTILE MATERIALS

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Indian Standard

METHODS FOR DETERMINATION OF WATER SOLUBLE CHROMATE IN TEXTILE MATERIALS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 December 1969, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

0.2 Water soluble chromium salts are used in dyeing of wool and other textile materials. If the water soluble chromium content of finished textiles is beyond a certain limit, it may cause some skin diseases when such textiles are worn next to the skin. The limit for water soluble chromium is, therefore, specified in many material specifications. It is hoped that this standard will be useful for determining the amount of water soluble chromium (expressed as sodium chromate or potassium dichromate) in textile materials.

0.3 The methods outlined in this standard is applicable only where the colour of the extracted dyes is destroyed by treatment with bromine.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS:2-1960*.

1. SCOPE

1.1 This standard prescribes methods for determination of water soluble chromium (expressed as sodium chromate or potassium dichromate) present in textile materials.

2. PRINCIPLE

2.1 A specimen is extracted with water and the amount of water soluble chromium is estimated volumetrically or by comparing the colour of the extract with the colour of the standard potassium dichromate solution.

*Rules for rounding off numerical values (revised).

3. SAMPLING

3.1 Sampling for Fibre and Yarn

3.1.1 Lot (Fibre or Yarn).—The quantity of fibre or yarn from the same source shall constitute a lot. If the lot contains more than 200 kg of fibre or yarn, it shall be divided in sub-lots each weighing 200 kg or less.

3.1.2 From a sub-lot 15 increments, each approximately weighing 10 g, shall be taken from different parts so that a representative sample is obtained. All the increments thus collected shall be thoroughly mixed. This shall constitute the **test sample**.

3.2 Sampling for Fabrics

3.2.1 Lot (Fabric).—The quantity of fabrics manufactured under relatively uniform conditions shall constitute a lot.

3.2.2 The number of pieces to be selected from a lot shall be as given below. The pieces thus selected shall constitute the gross sample.

<i>Lot Size</i>	<i>Sample Size</i>
Up to 100	3
101 „ 300	4
301 „ 500	5
501 and above	7

3.2.3 From each piece in the gross sample about 25 g of fabric shall be taken out from at least two different parts. The parts shall then be cut into further smaller pieces and thoroughly mixed. The pieces thus collected shall constitute the **test sample**.

4. CONDITIONING OF TEST SPECIMENS

4.1 Prior to test, the test specimens shall be conditioned for 24 hours to moisture equilibrium in a standard atmosphere at 65 ± 2 percent relative humidity and $27^\circ \pm 2^\circ\text{C}$ temperature (*see also* IS:196-1966*).

5. QUALITY OF REAGENTS

5.1 Unless specified otherwise pure chemicals shall be employed in tests and distilled water (*see* IS:1070-1960†) shall be used where the use of water as reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

*Atmospheric conditions for testing (*revised*).

†Specification for water, distilled quality (*revised*).

6. FIRST METHOD

6.1 Apparatus

6.1.1 Conical Flask

6.1.2 Beaker

6.2 Reagents

6.2.1 Mixed Acid Solution — prepared by mixing 150 ml of concentrated sulphuric acid with 150 ml of phosphoric acid and diluted to 1 litre with water.

6.2.2 Standard Potassium Dichromate Solution — 0.05 N.

6.2.3 Standard Ferrous Ammonium Sulphate Solution — 0.05 N.

6.2.4 Indicator Solution — prepared by dissolving 0.2 g of barium diphenylamine sulphonate in 100 ml of water.

6.3 Procedure

6.3.1 Take about 5 g of conditioned test specimen and weigh it accurately. Extract the test specimen with 200 ml of water (*see* Note 1) at $50^{\circ} \pm 2^{\circ}\text{C}$ for 2 hours with frequent stirring. Repeat the extraction once. Filter the extracts through a suitable filter paper (*see* Note 2). Wash the filter paper with water. Collect the two extracts and the washings into a beaker. Add 10 ml of mixed acid solution followed by 5 ml of ferrous ammonium sulphate solution. Add 1 ml of indicator solution and titrate against standard potassium dichromate solution until the first permanent blue colour appears.

NOTE 1 — The material to liquor ratio should be 1 : 40.

NOTE 2 — Whatman filter paper No. 42 or its equivalent is suitable for this test.

6.3.2 Carry out blank determination using distilled water and the same quantities of reagents.

6.3.3 Calculate the percentage of water soluble chromium (as sodium chromate) by the following formula:

$$P = \frac{(A - B) \times 0.27}{11}$$

where

P = percentage by weight of water soluble chromium (as sodium chromate),

A = volume in millilitres of potassium dichromate required for the blank (*see* 6.3.2),

B = volume in millilitres of potassium dichromate required for the test (see 6.3.1), and

W = weight in grams of the conditioned test specimen.

6.3.4 Repeat the test given in 6.3.1 and calculate separately the percentage of water soluble chromium (as sodium chromate) in each test specimen.

6.3.5 Calculate the average of the values obtained as in 6.3.3 and 6.3.4.

6.4 Report the value obtained as in 6.3.5 as the percentage of water soluble chromium (as sodium chromate) present in textile materials.

7. SECOND METHOD

7.1 Apparatus

7.1.1 Conical Flask

7.1.2 Nessler Tubes

7.2 Reagents

7.2.1 Dilute Sulphuric Acid Solution — 10 percent (w/v).

7.2.2 Buffer Solution — prepared by dissolving 9.90 g of boric acid and 3.81 g of sodium borate in sufficient amount of water and made up to one litre with distilled water.

NOTE — The buffer solution should be of pH 7.8.

7.2.3 Diphenyl Carbazide Solution — 0.2 percent solution (w/v) prepared by dissolving diphenyl carbazide [$\text{CO} (\text{C}_6\text{H}_5\text{NH-NH})_2$] in 9:1 mixture of rectified spirit and glacial acetic acid (v/v).

7.2.4 Standard Potassium Dichromate Solution — prepared by dissolving 0.01 g of potassium dichromate per litre.

7.3 Procedure

7.3.1 From the conditioned test sample, weigh exactly 2.5 g of test specimen. Cut it into small pieces and transfer them to the conical flask. Add 50 ml of buffer solution. Weigh the conical flask and note the weight. Maintain the contents of the conical flask at $50^\circ \pm 2^\circ\text{C}$ for 30 minutes with occasional shaking. Cool the flask to room temperature. Replace the loss in weight by addition of water. Filter the solution and collect the filtrate.

NOTE — If the extract is coloured, the colour should be destroyed by adding 2 ml of liquid bromine to the extract and then boiling the solution vigorously to evaporate off the bromine.

7.3.2 Take 20 ml of filtrate in a Nessler tube. Add to it 5 ml of dilute sulphuric acid and 2 ml diphenyl carbazide solution and make up the volume to 100 ml. Keep the contents in the flask for 10 minutes. Compare the colour developed with that of the solutions in which known quantities of potassium dichromate solution are taken separately instead of 20 ml of filtrate and treated similarly.

NOTE — A violet pink colour is developed with diphenyl carbazide.

7.3.3 Calculate the percentage of water soluble chromium (as potassium dichromate) by the following formula:

$$P = 0.001xv$$

where

P = percentage by weight of water soluble chromium (as potassium dichromate), and

v = volume in millilitres of standard potassium dichromate solution required to produce colour equal to colour of the extract.

7.3.4 Repeat the test with the remaining test specimens and calculate the percentage of the soluble chromium in each test specimen.

7.3.5 Calculate the average of the values obtained as in 7.3.3 and 7.3.4.

7.4 Report the value obtained as in 7.3.5 as the percentage of water soluble chromium (as potassium dichromate) present in the textile materials.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Conversion
Force	newton	N	1 N = 1 kg.1 m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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